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भारतीय मानक
लौह और इस्पात के लिए
फॉस्फेट लेपन की रीति संहिता
(पहला पुनरीक्षण)

Indian Standard
CODE OF PRACTICE FOR
PHOSPHATE COATINGS OF IRON AND STEEL
(*First Revision*)

ICS 25.220.60; 77.140.80

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The corrosion of metals is a vital problem. Considerable research has, therefore, been done and is still being done to find out suitable preventive measure against corrosion to suit the requirements of the individual users. Phosphating also known as 'parkerizing', 'granodizing', 'bonderizing', 'phosphatizing' and 'asfocoat' followed by sealing has been one of such protective schemes, which has proved to be economical and conveniently adoptable by various industries and miscellaneous users. The most wide-spread use of phosphate coatings is to prolong the useful life of paint finishes.

Phosphating is transformation of metal surfaces into new surfaces having non-metallic and non-conductive properties. They are widely used in the manufacturer of metal products for the following four principal reasons:

- a) To precondition metal surfaces for better bonding of paints and plastic coatings and to protect the surfaces against under-paint corrosion,
- b) To precondition metal surfaces for better retention of lubricants,
- c) To improve corrosion resistance by providing a good base for waxes and rust-preventing oils, and
- d) To prevent scuffing during bedding in sliding parts.

Indian Standard

CODE OF PRACTICE FOR PHOSPHATE COATINGS OF IRON AND STEEL (First Revision)

1 SCOPE

This code prescribes the details of the phosphate treatment process applicable to iron and steel.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS. No.	Title
245 : 1970	Trichloroethylene technical (<i>second revision</i>)
2074 : 1992	Ready mixed paint, air drying, red oxide-zinc chrome, priming (<i>second revision</i>)
2075 : 1979	Ready mixed paint, stoving, red oxide-zinc chrome, primary (<i>first revision</i>)
3531 : 1966	Glossary of terms relating to corrosion of metals
3618 : 1966	Phosphate treatment of iron and steel for protection against corrosion (<i>first revision</i>)

3 TERMINOLOGY

For the purpose of this code, the following definitions in addition to those given in IS 3531 shall apply.

3.1 Pointage

A conventional industrial measure of the strength (total acidity) of a phosphating solution. The pointage of a phosphating bath is the number of millilitres of 0.1 N sodium hydroxide (NaOH) solution (4.0 g/l) required to neutralize 10 ml of the phosphating solution, using phenolphthalein as indicator.

3.2 Toner Titration

It is the number of millilitres of 0.1 N potassium permanganate required to titrate 50 ml of the bath sample to a pink end point.

4 CLASSIFICATION OF TYPES OF COATINGS

4.1 The coatings produced differ in area, weight and or apparent density, depending on the type of material involved and the surface condition of the work pieces made from them, and depending on also on their mechanical and chemical treatment prior to phosphating, the composition of the phosphating bath

and the working conditions during phosphating. All phosphate coatings exhibits pores which can be largely sealed by after-treatment processes. Table 1 gives a survey of the various coating types.

4.2 Solutions based on $\text{Zn}(\text{H}_2\text{PO}_4)_2$, $\text{Fe}(\text{H}_2\text{PO}_4)_2$ or $\text{Mn}(\text{N}_2\text{PO}_4)_2$ yield zinc phosphate, ferrous phosphate (heavy) or manganese phosphating coatings respectively. With resolutions based on $\text{Zn}(\text{N}_2\text{PO}_4)_2 + \text{Ca}(\text{H}_2\text{PO}_4)_2$ or $\text{Zn}(\text{H}_2\text{PO}_4)_2 + \text{Mg}(\text{H}_2\text{PO}_4)_2$, zinc calcium phosphate coatings or zinc-magnesium phosphate coatings are obtained, respectively. Alkali phosphate solutions yield coatings consisting primarily of the phosphate of the treated metal mixed with oxides of the latter. These coating types are denoted by the following symbols:

Zinc phosphate	Znph
Zinc calcium phosphate	ZnCaph
Zinc-magnesium phosphate	ZnMgph
Manganese phosphate	Mnph
Ferrous phosphate	Fehph

4.2.1 When metal materials are treated in phosphating bath of which the characteristic constituent is, for example, NaH_2PO_4 (*see* Table 1), the following symbol applies:

Iron phosphate Feph

5 APPLICATIONS

5.1 Phosphate Coatings for Corrosion Protection

Coating types mentioned in Table 1 are suitable for controlling corrosion in many environments. The preferred coating type and the preferred coating weight are dictated by the type of metal to be phosphated and the requirements in respect of corrosion protection which are governed by the intended use of the phosphated parts.

5.1.1 Phosphate coatings for corrosion protection are usually given a final rinse with aqueous solutions containing chromium, or certain organic compounds, including tannins.

5.1.2 In the absence of additional treatment, phosphate coatings yield corrosion protection of only limited duration. For effective long-lasting protection to be achieved additional treatments adopted for the purpose of use of the phosphated metal surfaces are necessary, for example, the application of corrosion-protecting oils, greases or waxes or coating with paints, varnishes or similar coating materials (*see* Table 2 and Explanations).

Table 1 Coating Type of Phosphate Coatings
(Clauses 4.1 and 4.2.1)

Characteristic Constituent of the Phosphating	Coating Types Produced Coating	Appearance of Phosphate	Range of Area Weight of Coating Types			
			Ferrous Materials g/m ² (4)	Aluminium g/m ² (5)	Zinc g/m ² (6)	Cadmium g/m ² (7)
(1)	(2)	(3)				
$Zn(M_2PO_4)_2$	Zinc phosphate	Light grey to dark grey crystalline	1 to 60	1 to 15	1 to 60	1 to 60
$Zn(H_2PO_4)_2$ $Ca(H_2PO_4)_2$	Zinc-calcium phosphate	Light grey to dark grey, finely crystalline	1 to 15	—	1 to 10	—
$Zn(H_2PO_4)_2$ $Mg(H_2PO_4)_2$	Zinc-magnesium phosphate	Light grey to dark grey finely crystalline	1 to 15	—	1 to 10	—
$Mn(H_2PO_4)_2$	Manganese phosphate	Light grey to dark grey crystalline	1 to 60	—	—	—
$Me(I)H_2PO_4$	Phosphate of treated metal (plus iron oxides in the case of ferrous materials)	Amorphous coatings of 0.1 to approx 1 g/m ² : iridescent, for example, yellowish to bluish grey coatings over approx 1 g/m ² : grey	0.1 to <1	<0.3	0.1 to 2	—
$Fe(H_2PO_4)_2$	Ferrous phosphate	Dark grey crystalline	5 to 60	—	—	—

NOTE — Me(I) stands for cation of alkaline metal or NH_4^+ .

5.1.3 These additional treatment should preferably be carried out without long-term storage of the phosphated parts.

5.1.4 If phosphated metal surfaces are to be coated with paints, varnishes on similar coating materials, they must be virtually free from water-soluble substances deriving from the cleaning, treatment or rinsing baths and tending to cause bubbles to form afterwards in the film. The phosphate coatings are, therefore, rinsed afterwards with water which is free from salt and any other dissolved solids. Before such coatings are applied it is essential to avoid contaminating the phosphated metal surfaces, for example, with dust or finger prints.

5.2 Phosphate Coatings to Facilitate Cold Forming

Zinc phosphate coatings are preferred to facilitate cold forming. The area weight should be matched to the particular purpose (*see* Table 3), it is desirable in this case to neutralize the phosphate coatings after rinsing by treating with an aqueous weak alkaline solution.

5.3 Phosphate Coating to Facilitate Sliding Action

To facilitate sliding action, manganese phosphate coatings are generally preferred, but phosphate coatings of different composition and structure, such

Table 2 Phosphate Coatings for Corrosion Protection
(Clause 5.1.2)

Metal to be Protected	Phosphate, Coatings		Additional Treatment Needed	Protective Action Obtained	Examples of Application
	Preferred Coating Type	Preferred Area Weight			
(1)	(2)	(3)	(4)	(5)	(6)
Ferrous materials	Feph	0.1 to 1	None	Temporary corrosion protection in dry premises (no condensation)	Short-term in plant storage of machine components
	Znph	1 to 5			
	Znph	> 5	None	Long-term corrosion protection in dry premises (no condensation)	Long-term in plant of machine components
	Mnph	> 10			
	Znph	> 5	Corrosion protecting oils or waxes and the like, if necessary after drying of the phosphate coatings	Long-term protection in dry premises (no condensation) temporary corrosion protection in the open under a roof	Corrosion protection during long term storage and in service, for bolts, nuts, fittings, etc
	Feph	>10			
	Mnph	> 10			
	ZnCaph ZnMgph	> 5			
Ferrous materials, Zinc (where necessary), Aluminium, Cadmium	Znph	1 to 10	Paints, varnishes or similar coating materials	Long-term protection in the open and generally exposed to severe corrosion	Motor vehicle bodies, cabinets, sheets of refrigerators and washing machines
	ZnCaph	1 to 5			
Ferrous materials	Feph	0.1 to 1	None	Long-term protection in the open and generally when exposed to severe corrosion, particularly when bonding of the organic after-treatment coating is involved	Motor vehicle bodies, sheets and strips to undergo forming in the painted condition
Zinc	Znph	0.1 to 2	Paints, varnishes or similar coating materials		

Table 3 Zinc Phosphate Coatings to Facilitate Cold Forming
(Clause 5.2)

Type of Use	Preferred Area Weight, g/m ²
Drawing of steel wires	1 to 10
Drawing of welded steel tubes	1 to 10
Drawing of steel precision tubes	4 to 10
Cold extrusion	Above 10
Deep drawing without wall thickness reduction	1 to 5
Deep drawing with wall thickness reduction	4 to 10

as zinc phosphate coatings are also suitable. The type of phosphate coating to be used in a particular instance depends on the type of stressing involved.

5.3.1 The dimensional tolerances of the workpieces are the criterion for the choice of area weight (see Table 4). The phosphate coatings are used in conjunction with a suitable lubricant.

Table 4 Manganese Phosphate Coatings to Facilitate Sliding Action

Type of Use	Preferred Area Weight, g/m ²
Workpieces with small clearance fits, pistons of refrigerator compressors	1 to 3
Workpieces with large clearance fits, gears, crown wheels and pinions of gear units and differentials	5 to 20

6 METHODS OF APPLICATION

6.1 Immersion

Iron, zinc and manganese phosphate coating solutions may be applied by immersion. In accelerated baths, the treatment time varies between 3 to 30 minutes, while unaccelerated baths may take even 40 to 50 minutes. Baths requiring longer immersion periods generally produce greater thickness and crystal growth. Phosphating treatment should be continued till the completion of reaction as recommended by the manufacturer or until gasing ceases. Immersion processes are designed to give higher coating weight than those obtained by other processes.

6.2 Spray

Both the iron and zinc phosphate coating solutions may be applied by spray. The impinging action of the sprays makes it possible to clean and phosphate in less time than the immersion method at lower temperature (45 to 60°C). Spray applications seldom exceed 60 seconds, depending upon the weight of the coating desired. Spray application also develops finer and denser coatings. This method makes possible substantial saving in heating cost.

NOTE — Unaccelerated solution may be applied at nearing boiling temperature and accelerated bath according to the manufacturer's recommendations (usually at low temperature).

The principal advantages of low-temperature phosphating are:

- a) Savings in heating cost,
- b) Better working conditions,
- c) Safety to equipment, and
- d) Reduced sludge.

6.3 Steam or Air Spray

When surfaces and objects are too large to be processed

through spray washing machines or immersion in a solution tank or the volume of production does not warrant installation of such equipment, phosphate coatings may be applied through solution lifting guns using steam or compressed air. Basically, the gun is an extended venturi tube system. As steam or air passes through the venturi, the vacuum created draws the phosphate solution into the flow which passes through the exit nozzle of the gun over to the surface to be treated. It has the physical force and chemical activity desirable for phosphating of the parts. When steam is used, the process has the extra advantage of heat.

6.4 Brush

Large components, machinery parts and structural parts which can not be phosphated by immersion method, may be phosphated by brushing method. However, care shall be taken to ensure that all rust, scale, grease and other contaminations are removed before application of the phosphating solution.

7 EQUIPMENT

7.1 The requirements of the plants for phosphating will depend upon the method of phosphating process and shall be furnished by the supplier of the phosphating materials. However, the following equipment are generally needed:

- a) Degreasing plant (trichloroethylene or alkali degreasing tank with rinse);
- b) Rust or scale removing solution tank;
- c) Cold water tank (with constant water overflow);
- d) Phosphating tank of stainless steel, heavy mild steel or rubber-lined construction, heated by gas, steam or electricity;
- e) Cold water rinse tank (with constant water overflow);
- f) Hot-chromate dip tank (containing hot chromic acid at 60 to 80°C);
- g) Drying oven or hot plate, if necessary; and
- h) Sealing tank.

7.1.1 Care should be taken to avoid the use of unsuitable material in plant construction, for example, copper or brass heating coils, which adversely affect the quality of phosphate coating formed.

7.1.2 The size of the installation will depend on the size and number of articles to be processed and may vary from a small hand-operated unit of about 4 000 l capacity to a large fully automatic conveyor type. A diagrammatic layout is shown in Fig. 1.

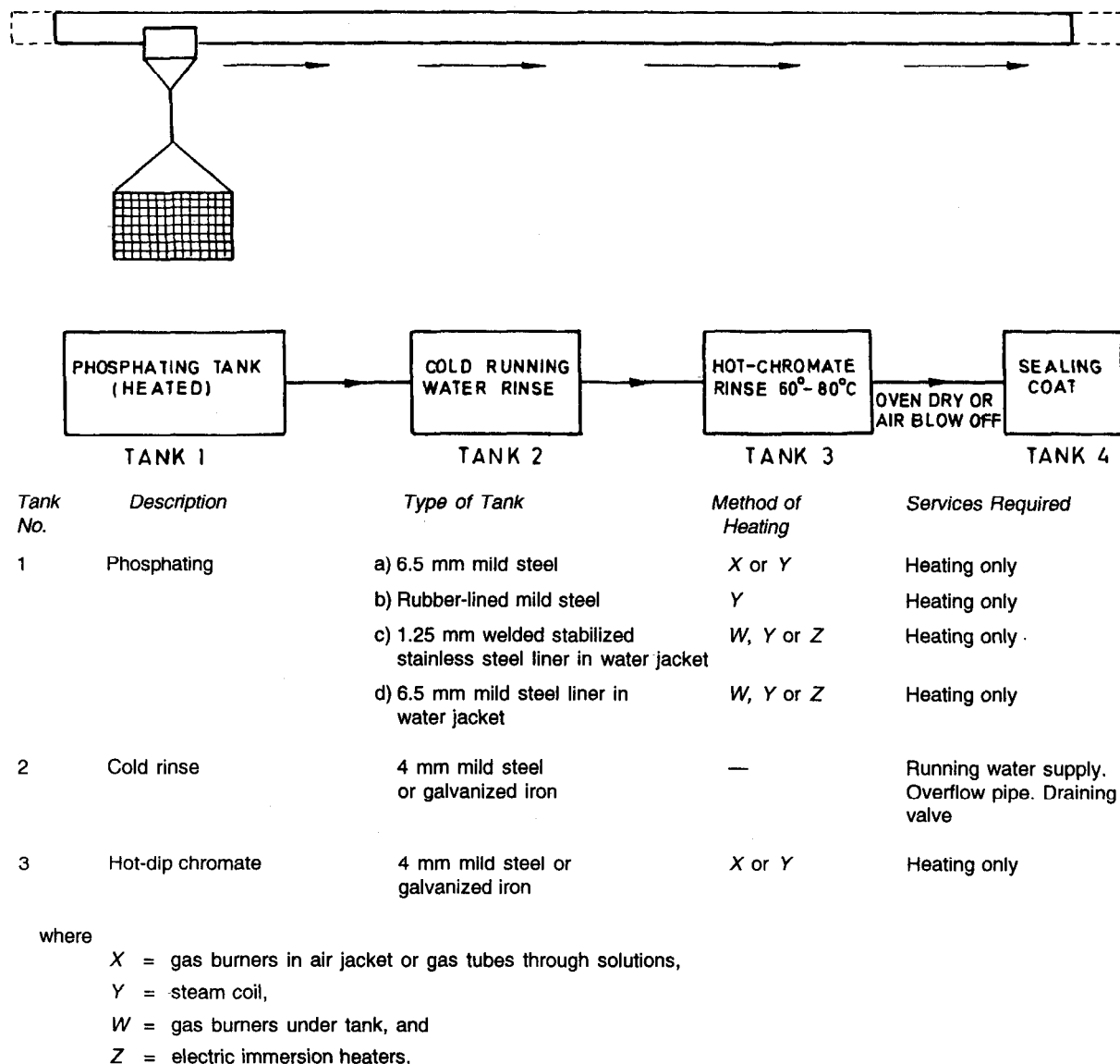


FIG. 1 LAYOUT FOR PHOSPHATING PROCESS

7.1.3 Phosphating Tank

The phosphating tank may be made of mild steel approximately 6 mm thick of welded construction. Stainless steel, however, gives longer life. The dimensions of the tank will depend on the output of work required, but should be kept as small as possible consistent with ease of working. However, the depth of the tank should be sufficient to allow for accumulation of precipitate and normally not less than 1 500 mm. It will be better if a false bottom is also provided at about 100 mm above the bottom. Mild steel hooks, trays or wire baskets should be used to suspend articles in bath. Draining valves in the bottom of the tank are not recommended because of their tendency to clog or leak. A hinged lid is advantageous for reducing the amount of steam emitted from the tank. A wall-mounted thermometer is required to facilitate temperature control.

7.1.3.1 Heating of phosphating tank

The following three methods are generally used for heating phosphating tank:

- Gas* — The most suitable method of heating is by gas burner placed beneath the bottom level of the tank and to the side of it so that the flame is directed between the tank and an outer insulated jacket. It is important that the flame does not come in contact with the bottom of the tank, otherwise the precipitate will be disturbed and will be deposited on the metal being processed. Alternatively gas burners may be placed in tubes which pass through the solution about 150 mm from the bottom of the tank.
- Steam* — For this method of heating, a mild steel/stainless steel coil is required and should

be placed at the side of the tank clear of precipitate. It should not be placed on the bottom of the tank. The steam connections should enter over the side of the tank with flanged bolted coupling to facilitate removal of the coil for cleaning purposes. A live steam line may be used as an auxiliary source of heating by placing it in a horizontal position about halfway down the side of the tank with 1.5 mm holes drilled at about 25 mm intervals along the top surface to avoid disturbing the precipitate. When steam coil is in direct contact with the solution, phosphating tanks rapidly scale up, the trouble will usually be found to be due to the steam pressure being too high resulting in extreme local heating, which promotes rapid and heavy scale formation. For this reason a pressure reducing valve is required to limit the steam pressure to 1.8 to 2.1 kgf/cm². When a steam coil is used in a water jacket, the problem of rapid scale formation does not arise.

Therefore, no pressure reducing valve is necessary.

- c) *Electricity* — A jacketed tank can be heated by electric immersion heaters placed in the jacket. The jacket should be filled with oil. Alternatively, the heater may be immersed in a small oil jacket which is itself immersed in the solution. Under no circumstances shall the heater be immersed direct in the solution as it would rapidly scale up and become inefficient. Low-voltage rod heaters operated from transformer may be used. The heating requirements of the tank will depend upon the size and the efficiency of the lagging; the details of the equipment will be supplied by the supplier of the phosphating.

7.1.4 Rinse Tank

These should be made of mild steel or galvanized iron, similar in size to the phosphating tank. The cold rinse tank require a continuous supply of water and overflow and a draining valve. The hot chromate rinse tank requires a steam coil or other heating unit together with a water supply, scum through overflow and a draining valve.

SECTION 1 SURFACE PREPARATION

8 GENERAL

Surface preparation is essential prior to phosphating for obtaining a satisfactory protective coating. Before processing, all scale, rust, grease, oil and foreign matter shall be removed from the surface to be treated by a method or a combination of methods suitable for the particular phosphating process. Where necessary, the cleaned components shall be adequately rinsed in cold or hot water to remove such residues of cleaning materials as might affect the bath or the quality of the coating. Surface preparation could be

broadly classified in three groups as described in 9, 10 and 11.

9 REMOVAL OF OIL, GREASE, DIRT AND SWarf

9.1 Surface cleaning in this group relates to complete removal of oil, grease, dirt and swarf from unit parts or assemblies. Several methods of processing are available, choice of the appropriate process depends upon the type and degree of contamination and the size and shape of the parts.

9.1.1 Petroleum-Solvent Cleaning

9.1.1.0 These methods relate to the use of petroleum solvents such as kerosene and mineral turpentine for the initial removal of heavy deposits of grease, dirt and swarf from units parts or simple assemblies having easily accessible surfaces. Petroleum solvents may also be applied to the *in-situ* cleaning of large units, assemblies or machinery which cannot be accommodated in degreasing equipment. Petroleum solvent should not be used for degreasing equipment. Petroleum solvent should not be used for assemblies containing fabric, rubber, or other non-metallic materials, unless it is known that no harm will result. Petroleum solvent cleaning suffers from the following drawbacks:

- Brush, wipe or immersion methods being manual in operation are slow and time consuming and, therefore, unsuitable on their own for continuous production lines.
- The degree of cleanliness achieved by such methods is not high and they are generally used for preliminary cleaning to supplement a proper degreasing operation.

9.1.1.1 Procedure for petroleum-solvent cleaning

- Processes* — The two main methods are outlined below. Precise details depend upon the quantity and characteristics of the parts or assemblies to be cleaned:
 - Immersion* — This method may be used for parts which are conveniently handled in small tanks. The articles should be immersed in the solvent long enough to allow removal of the contaminants, and in no case for less than one minute. Agitation is desirable and brushing or scrubbing will aid and speed cleaning. Articles with cavities that may hold the solvent should be immersed so that the holes are filled and then removed at an angle which will ensure that they are emptied. Repeated dipping and agitation may be necessary to flush out solid materials such as swarf.

The articles should then be immersed in a second tank of clean solvent (agitated if possible) for at least one minute to remove the film of contaminated solvent from the first tank. Care should be taken to carry over

as little solvent as possible from one tank into the other.

- ii) *Brushing or wiping* — This method is intended for the *in-situ* removal of oil and grease, from articles which cannot be conveniently handled by conventional immersion or spray methods. It can also be used for the selective degreasing of certain areas of painted assemblies which might be damaged by general application of solvent. It may also be used for the *in-situ* cleaning of articles too large for immersion tanks or spray cleaning systems.

Petroleum solvent should be applied to the contaminated areas with a clean brush or a cloth soaked in the solvent, the application of clean solvent with scrubbing or wiping should be repeated until all the contamination has been removed. Care should be taken to apply the solvent to the contaminated metal areas only.

- b) *Handling precautions* — Articles should be placed on hooks or racks or in suitable containers that permit adequate draining. They should not be handled with bare hands after cleaning, clean gloves or similar protection, should be used and handling kept to a minimum.
- c) *Safety precautions* — Petroleum solvents should be used at room temperature and suitable fire extinguishers should be provided, as these solvents are inflammable.

Oil-resisting synthetic rubber gloves should be worn when handling articles during cleaning, to protect the articles from sweat residues and to avoid any possible affect of the solvent on the skin. Care should be taken to avoid breathing the petroleum solvent vapour.

9.1.2 Trichloroethylene Cleaning

9.1.2.0 This method relates to surface cleaning with the solvent trichloroethylene conforming to IS 245. Trichloroethylene degreasing should not be used on assemblies containing fabric, rubber or other non-metallic materials unless it is known that no harm will result.

9.1.2.1 Procedure

- a) *Degreasing* — The three main processes are described below. The precise details of the equipment and the method of operation for each process depends on the quantity and characteristics of the parts or assemblies to be cleaned; the plant manufacturer's recommendations should be followed.

- i) *Vapour process* — To remove simple films of oil and grease, articles may be subjected to the vapour process, in which the parts are exposed in a bath of solvent vapour; the vapour condenses on the cold surfaces of the articles and the condensate dissolves the oil

and grease; taking it away to the base of the tank. To ensure the maximum condensation the temperature of the articles should be near to room temperature as possible at the time of immersion; they should be passed through or suspended in the solvent vapour until no further condensation occurs, after which no further degreasing will take place. Articles with a very heavy film of grease may need a second exposure after cooling.

- ii) *Liquid process* — Loosely-bound contamination (too much for vapour treatment), such as polishing compound, swarf and road dirt can be removed by immersing the parts in vigorously boiling trichloroethylene. The articles should be passed through or suspended in the boiling solvent until all oil and dirt have been removed. Heavily contaminated articles should be immersed in two or even three separate compartments containing progressively clean solvent. Immersion in one bath of boiling trichloroethylene may be followed (after cooling) by exposure in the solvent vapour until no further condensation occurs.

- iii) *Jetting process* — Articles with obstinate dirt deposits that cannot be removed by boiling trichloroethylene may require jetting at high pressure with the hot solvent. The jetting should be carried out only in equipment specially designed for the purpose.

- b) *Drying* — The articles after trichloroethylene — degreasing are hot and normally dry.
- c) *Handling precautions* — During processing, the articles should be placed on hooks, or racks or in a suitable container. They should be so arranged that there is adequate opportunity for the solvent to drain from holes, crevices and other irregularities, parts or assemblies that could trap solvent should be rotated or tilted during the degreasing process to prevent drag-out of trichloroethylene. Assemblies that would trap solvent even though rotated to tilted should either be dismantled before cleaning or be so handled that cleaning is not necessary after final assembly. Handling can be reduced by using mechanical and conveyer type degreasing plants which automatically rotate or tilt the articles. After cleaning, articles should not be handled with bare hands, clean gloves or similar protection should be used.
- d) *Safety precautions* — Trichloroethylene is a toxic substance, care should be taken to avoid breathing the vapour near degreasing equipment. No person should enter pit or vessel which contains trichloroethylene or in which the solvent may be present. Trichloroethylene is non-inflammable but naked flames may cause decomposition of the solvent vapour with the production of harmful acidic gases and they should not be allowed near

degreasing equipment. Though trichloroethylene is excellent solvent or mineral and vegetable oil, sometimes materials like lanolin are not completely removed. Of late perchloroethylene instead of trichloroethylene is widely used as this has higher boiling point which means longer condensation period on light-gauge work with superior degreasing effect.

9.1.3 Alkaline Cleaning

9.1.3.0 This method relates to the use of alkaline cleaning solutions for the complete removal of oil, grease, dirt and swarf from unit parts or simple assemblies having easily accessible surfaces. Alkaline cleaning should be used with caution on articles with highly finished surfaces. Porous articles or parts and assemblies that would trap the solution should not be cleaned with alkaline cleaners owing to the difficulty of rinsing away all traces of the solution, and of drying.

9.1.3.1 Method of alkaline cleaning

a) *Processes* — The three main methods are described below but precise details of the equipment and method of operation for each process depend upon the quality and characteristics of the parts or assemblies to be cleaned. The recommendations of the supplier of material and equipment should be followed. Immersion cleaning is by far the most commonly used method for degreasing. Spray treatment is followed where the value of production is high, or the contamination is very stubborn, or both. electrolytic cleaning is seldom used (if ever, before phosphating) since it is costly and the degree of cleanliness achieved by other methods is adequate for the needs of the phosphating treatment.

b) Immersion process

i) *Tank arrangement* — This process is easy to operate, and a diagrammatic layout of a simple alkali-degreasing tank is shown in Fig. 2.

The tank which can be constructed from mild steel should have an overflow weir to a small separate compartment. The surface of the alkaline solution can then be kept free from scum by directing across it, from a point opposite the weir, a stream of solution pumped continuously from overflow compartment. A ball-float water supply valve should be fitted above the pump suction in the overflow compartment, so that evaporation losses are automatically replaced. The compartment shall, of course, be big enough to hold the solution displaced by introduction of a load of work.

It is always desirable to ensure that the degreasing solution is agitated either by convection currents from a heating unit at one end of the tank or by some other means, so that there is a continuous movement of solution over the surface of the work.

ii) *Method of use* — Alkaline cleaners shall be used at as high a temperature within the range 90 to 95°C to obtain the best results as possible. The concentration of the solution varies with the type and degree of contamination, and also the immersion time allowed for degreasing. Lightly contaminated objects may require about 10 minutes at 4 percent concentration, but objects with heavy or stubborn contamination may require 10 minutes or more at higher concentrations.

The alkaline mixture should be well stirred

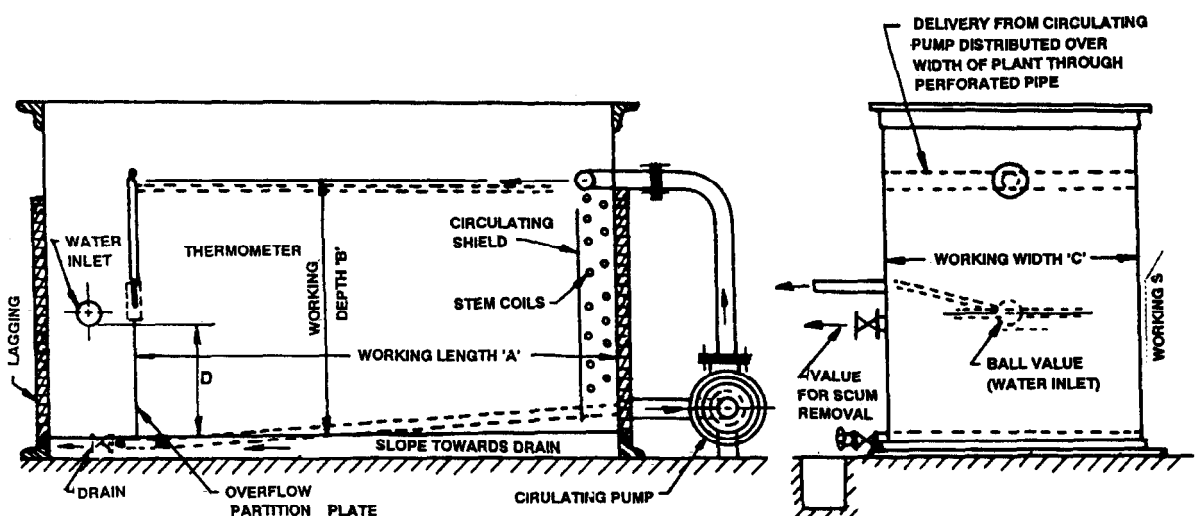


FIG. 2 DIAGRAMMATIC LAYOUT FOR SIMPLE ALKALI-DEGREASING PLANT

into hot water when making up a new bath. If a larger degreasing tank is used, it is desirable to carry out the mixing in a small tank provided with stirrer or paddle.

Thorough rinsing in water shall follow the cleaning stage. Articles should be allowed to drain for a few seconds on withdrawal from the alkali tank, to minimize carry out into the rinse tank but the time should not be so long as to allow the cleaning solution to dry on the parts. Running water should be provided for the rinse tank and sufficient rinsing time allowed for the complete removal of the cleaning solution; if rinse in hot water (90 to 95°C). Skimming arrangements similar to those on the alkaline solution tank may be used to ensure the cleanliness of the water in a hot rinse tank.

- iii) *Composition of alkaline cleaners* — Chemical cleaning is usually carried out in aqueous solutions of alkalis such as caustic soda ash, phosphates, and silicates with the sodium salts being preferred because of their lower cost. These chemicals exert different effects on the various contaminants and have to be blended in well defined proportions to achieve optimum results. All alkaline cleaners contain surface active agents, which are present in small quantities, but exert a very decisive influence on degreasing efficiency.

Examples of a few typical cleaners formulations are given below:

- 1) Heavy duty cleaner used at near boiling point:

	Percent
Caustic soda	53
Soda ash	36
Trisodium phosphate	9
Wetting agent	2

- 2) Light medium duty cleaner used at near boiling point:

	Percent
Sodium metasilicate	97
Wetting agent	3

- iv) *Testing of alkaline solutions* — To achieve consistently high degreasing efficiency, it is necessary to test the degreasing batch regularly and maintain it at the recommended concentration. This is achieved by a simple titration in which 20 ml of the batch solution are titrated against normal solutions of hydrochloric or sulphuric acid using methyl orange as indicator. The strength of the degreasing bath may be approximately calculated by dividing the number of millilitres of normal acid used by (3).

- c) *Spray-degreasing process* — For unit parts or simple assemblies with oil and obstinate dirt deposits alkaline jet cleaning may be used.

Articles should be placed in suitable apparatus and subjected to jets of hot alkaline solution at high pressure.

The conveyer of continuous jet washing machines should be loaded so as to permit the jets of solution to reach all surfaces of the articles being cleaned.

Alkaline cleaners are used in jetting or spraying machines at concentrations lower than those for an immersion process and concentrations of the order of 1.5-2.0 percent are generally recommended.

The temperature of the jetting solution depends on the design of the machine and the time over which the work is cleaned. Generally 75 to 80°C is a suitable temperature range.

After jetting components should be thoroughly washed and the final rinse should be in hot after, if parts are required dry after treatment. Where thin gauge material of low heat capacity is used, it may be necessary to have a hot-air drying arrangement at the exit end of the machine.

As for immersion cleaning, it is necessary to test the spray degreasing chemical regularly and maintain it at the recommended strength. The testing method is identical to one given for immersion cleaning [(see 9.1.3.1(b))].

- d) *Electrolytic cleaning* — Alkaline electro-cleaning may be used for articles that cannot be cleaned by simple immersion methods. It is quicker and more effective in removing adhering solids. The components should go to electro-cleaning in a reasonably cleaned state. For this, they should receive preliminary treatment to remove oil, grease, paint and loose rust.

The steel tank containing the electrolyte or mild steel in any suitable form shall be used as one electrode (anode or cathode). The component to be degreased is made the other electrode. Graphite may be used as an alternative anode material. This will need greater care in handling because of its brittle nature but possesses the advantage of being unattacked. Normally the article to be cleaned is made cathode as grease removal is more rapid and efficient than when it is made anode. However cathodic treatment may entail harmful effects from hydrogen absorption, anodic treatment is therefore preferred. The treatment may be done over a range from room temperature to near boiling temperature. Higher temperature facilitates quicker cleaning.

If two tanks with current-reversing switches are provided, articles for which cathodic cleaning is permissible, should be cleaned as the cathode for 1 to 5 minutes and then as the anode for 15 to 30 seconds. If only one tank without current-reversing switches is available, articles should be cleaned for 1 to 5 minutes as the anode only.

- e) *Rinsing* — All alkaline cleaning should be followed by adequate draining but the drainage time should not be so long as to allow the cleaning solution to dry on the articles. Water rinsing should follow draining to ensure that carry-over of alkali to the next stage is kept to the minimum. The work should not be allowed to dry before rinsing. The first rinsing may be given by water jets, particularly in the case of jet-washing machines.

It is preferable that articles be immersed in rinse tank with running water and that this be followed by immersion for 30 to 60 seconds in hot water (80 to 95°C) so that subsequent drying is facilitated. In the final rinse tank, there should be sufficient flow to keep the water clean and to remove surface scum, agitation may be desirable.

Slightly alkaline rinse water (provided that the alkaline does not exceed 0.1 percent) prevents steel from rusting during drying.

- f) *Handling precautions* — During processing, the articles should be placed on hooks or racks or in suitable containers; for electro-cleaning these supports should maintain good electrical contact and be adequate to conduct the current. Articles should not be handled with bare hands during and after cleaning. Cleaned gloves should be used and handling kept to a minimum.
- g) *Safety precautions* — Strong alkalis attack the eyes and the skin, and therefore, goggles, rubber gloves and suitable protective clothing should be worn when handling alkaline mixtures of their solutions.

9.1.4 Emulsion Cleaning

9.1.4.1 These are either single or diphase cleaners of oil-water emulsion type. They contain very little alkali but rely on powerful emulsifying agents and hydrocarbons to remove grease. While they cannot be compared with heavy duty cleaners based on caustic soda, they are very satisfactory for the most of cleaning jobs. Emulsion cleaning is particularly preferred before phosphating as it subsequently leaves a fine textured phosphate coating. Besides, its operating temperature is generally lower than that of heavy-duty alkaline cleaners. Emulsion cleaners may be applied both by spray and dip, though the former is more common.

Emulsion cleaners should be correctly formulated otherwise emulsion stability will pose problems. There are several proprietary products and manufacturers instructions should be followed for their operation to get the best results.

9.1.4.2 Equipment

Emulsion cleaners may be used in simple mild steel tanks for dip application or mechanized power-spray plant for spray application. Heating may be done by steam, electricity or gas.

9.1.5 Steam Cleaning

9.1.5.0 The method relates to the use of a jet of high pressure steam for the *in-situ* cleaning of large unit parts, assemblies and machinery that cannot be accommodated in a cleaning apparatus. The cleaning may be carried out with pure steam, or with alkaline solution and steam mixtures.

9.1.5.1 Method of steam cleaning

- a) *Cleaning* — Where a steam supply is available, it may be used in conjunction with a simple steam gun, or with an injector that will entrain alkaline solution into the steam jet, to remove oil and dirt contamination from the whole surface of the article. Straight steam will remove most of the oil and grease, and loose-adhering dirt, alkaline solution and steam mixture will readily remove closely-adhering dirt as well as oil and grease.

The alkaline solution should be at a concentration of 2 to 4 percent. Special steam cleaning equipments are available which produce steam at pressures up to 10 kgf/cm² within a few minute of starting from cold and they permit an immediate changeover from alkaline solution and steam to straight steam or *vice-versa*.

- b) *Rinsing* — On completion of alkaline solution and steam cleaning straight steam should immediately be directed over all cleaned surfaces so as to wash away alkaline deposits.
- c) *Handling precautions* — During processing, the articles should be positioned to allow draining of the steam condensate. Care should be taken to ensure that steam does not enter delicate mechanisms, armature windings or other working parts from which water cannot easily be removed.

The handling of articles during cleaning should be kept to a minimum and sweat contamination avoided by the use of clean gloves.

- d) *Safety precautions* — Goggles and protective clothing should be worn when carrying out steam cleaning with strong alkalis, particularly in confined places. Due care should be taken to avoid steam burns.

10 REMOVAL OF RUST AND SCALE

10.1 Rust may be present after degreasing owing to exposure to corrosive conditions during manufacture. Owing to its hygroscopic nature, rust retains a certain amount of water, and further corrosion may thus occur underneath a deposit of rust. It is, therefore, necessary that rust be removed before applications of phosphate treatment.

Scale may be present from operations during manufacture (for example, hot rolling, forging, welding or heat treatment). Both rust and scale invariably cause protective coatings to fail prematurely.

Rust and scale may be removed by any one of the methods described under 10.2.1, 10.2.2, and 10.2.3 or by a combination thereof.

10.2 The choice of the method of de-scaling or de-rusting depends on the character and thickness of the deposit to be removed, size, shape, material and construction of the article and the finish required. For example, the removal of heavy mill scale requires prolonged pickling in acid or heavy shot blasting, it cannot be effected by scratch brushing. On the other hand the number of articles is large, by a dilute-acid pickle. Special methods of pickling may be required for high alloy steels. In general, the least severe treatment necessary to attain the desired result should be used.

10.2.1 Mechanical Cleaning Methods

10.2.1.1 Hand cleaning

Hand cleaning is a method of preparing metal surfaces by removing loose mill scale, rust and paint by hand brushing, sanding, sand scraping, hand chipping or other hand impact tool or by a combination of these methods. Stratified rust (rust scale) shall be removed by hand hammering, chipping other hand impact tools or a combination of them. All loose mill scale and loose and non-adherent rust shall be removed by hand wire brushing sanding, scraping, or by combination of these methods. All accessible weld flux and spatters shall be removed by hand scraping or by hand-impact tools followed by wire brushing. All rivets, welds, corners, joints and openings shall be properly cleaned. The steel wire of the wire brushes shall have sufficient rigidity to clean the surface and shall be discarded when they are not longer effective. Hand scrapers shall be made of suitable material and shall be kept sharp enough to be effective. The tools shall be operated in such a manner that no burst or sharp ridges are left on the surfaces and no sharp cuts are made into the steel. After hand cleaning is completed, dust and other loose matter shall be removed from the surfaces. If detrimental amount of grease or oil are still present, these areas shall be spot cleaned with solvents.

The labour involved can be reduced and the results improved by using power-driven tools. A wide variety of these has become available in recent years, such as grinding wheels, chippers, scrapers, needle gun and wire brushes.

Limitations of this method are (1) time and labour consuming (2) do not yielding a chemically-clean surface (3) while commonly adopted for cleaning of structures (which are not phosphated anyway), it is not amenable for continuous production lines or for large volume of production.

10.2.1.2 Blast-cleaning (or blasting)

Blast-cleaning is a method of preparing metal surfaces by removing all scale, rust, paint or foreign matter by the use of abrasives propelled through nozzles or

centrifugal wheels. Small quantities of oil or grease may be removed by the blast-cleaning operating. If oil and grease are removed by blast-cleaning, the abrasive shall not be re-used, if such re-use is detrimental to the surface. The surface of the metal can be blast-cleaned by any of the following methods:

- a) Dry sand blasting is done to protect the surface of the workpiece from metallic contamination using compressed air, blast nozzles and dry sand, garnet, novaculite, dolomite, pumice, flint, quartz or manufactured materials, such as aluminium oxide, silicon carbide and slag, of a minimum particle size not more than that passing through 1 mm IS Sieve screen. The natural materials are lowest in initial cost and the manufactured materials, although somewhat more expensive than natural sands, cost less than metallic abrasives.
- b) Grit blasting using compressed air, blast nozzles and crushed grit made of cast iron, malleable iron, steel or synthetic grits other than sand of a maximum particle size or not more than that passing through 1 mm IS Sieve.
- c) Shot blasting using compressed air nozzles and cast iron, malleable iron, steel or synthetic shot of a maximum size not more than that passing through 1 mm IS Sieve. Shots may be made of aluminium or cut steel wire.
- d) Blast-free circulating nozzles using compressed air or vacuum and any of the preceding abrasive.
- e) Grit or shot blasting using centrifugal wheels and crushed grit made of cast iron, malleable iron, steel or synthetic grit of a maximum particle size not more than that passing through 1 mm IS Sieve.

The choice for various blasting processes will be governed mainly by convenience and cost. Particular care is necessary to adjust the operating conditions when blasting heavily rusted steel. Otherwise the abrasive may driven the rust into the pits instead of removing it from them. Moreover, it is always essential to dust down the surface after blasting by brushing or vacuum cleaning or an air blast.

Shot or sand blasting or abrasive treatments should not be applied to finish machined parts, but are valuable for use on articles immediately after fabrication or heat treatment when hard scales are often present. Shot blasting produces a coarser finish than sand blasting causing silicosis due to silica dust, but involves less hazard to the health of the operator.

10.2.2 Chemical Treatment Methods

10.2.2.1 Sulphuric or hydrochloric acid pickling

Pickling with these acids is of particular value in descaling, but should be used only for parts that can

be easily and thoroughly washed free from acid. It may thus be unsuitable for parts of complicated shape. Particularly those containing narrow channels or blind holes that cannot be properly washed out, or parts with porous surface layers. The following types of components, therefore, should not normally be treated with these acids:

- a) Parts built up by riveting, spot welding, or similar methods;
- b) Cast-iron parts, owing to the possibility of occlusion of pickling acid in porous surface layers, or with machined cast iron in graphite pockets;
- c) Ferrous articles with associated non-ferrous or non-metallic parts because of the risk of attack of these parts due to electrolytic effects, and acid may be trapped at the joint; and
- d) In case of high strength steel having strength more than 1 000 MPa, which are susceptible to hydrogen embrittlement and pickling may be avoided.

For the operation of sulphuric or hydrochloric acid pickling, various types of acid-resistant tank are available, for example, lead (for sulphuric acid), glass or glazed earthenware; wood, steel or concrete lined with rubber or other acid-resisting materials may also be used.

Several efficient inhibitors to reduce acid attack on the base metal are available commercially and one of these should be employed; it is necessary to adhere strictly to the manufacturer's instructions regarding suitability, concentration and method of addition.

In hydrochloric acid pickling the concentration of acid may be varied between 1 to 50 percent of concentrated hydrochloric acid by volume, according to the nature and amount of scale or rust and the time available for pickling. Higher concentration of acid removes rust and scale more rapidly but may attack the steel more severely. Hydrochloric acid pickles work reasonably well without external heating; after the heat of reaction between the acid and the scale is sufficient to keep the bath at 30 to 40°C and then quite rapid pickling takes place.

In sulphuric acid pickling the concentration may be between 5 to 20 percent of sulphuric acid by volume and the bath is preferably heated (for example, by steam coils) to a temperature of about 60 to 85°C. It is very uneconomical to pickle in other than hot-sulphuric acid solutions because of the relative slowness of the attack.

NOTE— Where sulphuric acid solution are employed, the usual precaution should be taken to avoid accident from overheating when it is mixed with water, that is, the acid should be added slowly to the water, and not vice-versa.

Pickling may be accelerated to some extent by mechanical agitation of the parts or of the solution, and sometimes by lightly scrubbing off deposits that have loosened in the acid bath. Completion of pickling is best judged by periodic inspection.

The acid content of the bath should be checked frequently and additions of acid made to maintain the correct strength. The pickle should be discarded when the iron content has risen to such an extent as to retard seriously the rate of pickling. For hydrochloric acid pickle, the limiting iron content is 120 g/l and for sulphuric acid pickle 80 g/l.

After removal from the sulphuric and hydrochloric acid pickling bath, the parts should be thoroughly washed in running water to remove all traces of acid. After removal from the solution, parts should be thoroughly rinsed in clean hot water with a very small alkali content and dried.

10.2.2.2 Phosphoric acid pickling

While phosphoric acid is more expensive than sulphuric acid, it involves less danger of corrosion from residues or during drying and is, therefore, preferable to the other mineral acid, nevertheless, with certain exceptions, it is necessary to wash the articles thoroughly after the pickling treatment, particularly, if the parts are of complicated shape, contain narrow channels or blind holes or are built up by such methods as riveting or spot-welding.

10.2.2.2.1 Rust removal with phosphoric acid

Light rust may be removed by immersion in cold phosphoric acid or in proprietary liquids based on phosphoric acid and substantially free from other mineral acids, diluted for use according to the manufacturer's instructions (these liquids may contain wetting agents' to facilitate action on slightly greasy surfaces). The optimum strength of acid is approximately 25 percent by volume. Generally, the procedure is as follows:

- a) *Dip application* — Immerse in the rust-removing solution, if necessary assisting the de-rusting action by brushing with a steel wool pad. Alternatively, warm the solution to 60°C to speed up rust removal. The immersion should not be longer than is required for complete de-rusting; normally up to an hour (or 15 minutes at the higher temperature) should suffice. Lead or rubber-lined tanks are recommended for the rust-removing solutions.

The parts should not be dried prior to phosphating. Further rinsing of the articles either in cold running water or preferably in hot water should be carried out to ensure thorough removal of acid residues, if any. The rinse water should at all times remain clean and should not exceed a contamination limit of 1 percent of the strength of the previous stage.

If composite articles are to be treated, care should be taken to avoid excessive attack on non-ferrous metals. Generally the solution should not be used for thin leaf springs or springs under stress. Normalized, locally hardened or hardened and tempered steels, and spring steels should be given

a further treatment for 30 minutes in boiling water. Sodium chromate in the proportion of 1 g/l may be added to this water as a rust inhibitor.

- b) *Brush applicator (light rust)* — Apply the rust remover with a brush or swab, rubbing where necessary with a steel-wool pad to assist rust removal. Keep the surface well wetted with rust-removing solution. A long-handled brush minimizes the likelihood of hands or clothing being splashed. Wash off the solution thoroughly after de-rusting paying particular attention to seams and crevices.

10.2.2.2.2 Scale removal with phosphoric acid

Heavy scale may be removed by phosphoric acid only at higher temperature, for example, at 85°C for 25 percent acid (v/v). It is not generally necessary to use an inhibitor in the bath. After pickling is complete, the treatment of the articles by washing, drying, etc., is the same as in rust removal. The bath should be discarded when the concentration of iron reaches 2 percent, or powdery deposits may be formed on the metal surfaces.

10.2.2.3 Duplex sulphuric and phosphoric acid process

Economy in the use of phosphoric acid may be effected by the use of the Footner's process for de-scaling steel plate and other forms of structural steel prior to the application of phosphate coating. The process consist of:

- a) Pickling in 5 to 10 percent sulphuric acid (v/v) at 60 to 65°C in presence of an inhibitor for 12 to 15 minutes or until all scale and rust is removed. Further sulphuric acid should be added when the pickling time increases appreciably. The bath should be discarded when the accumulation of sediment, etc., and the concentration of iron in the solution interfere with the pickling and result in deposits on the surface of the article. This occurs when the specific gravity reaches about 1.18 to 1.20 or there is 1.6 percent of iron in the solution. After the article is lifted from the acid bath it should be allowed to drain for 15 to 30 seconds before immersion in the water-bath.
- b) Washing in warm water (60 to 65°C) by immersing twice before passing to the final bath. There should be a small flow of water through the water-wash-bath to prevent the total acidity, determined by titration with phenolphthalein, from exceeding 0.1 of sulphuric acid per 100 ml. The necessary flow of water may be established after a short experience of the process.
- c) Immersing for 3 to 5 minutes in 2 percent phosphoric acid solution maintained at a minimum temperature of 85°C. When the iron content exceeds 0.5 percent, a proportion of the bath should be discarded and the bath replenished by suitable additions of clean water and phosphoric acid.

When removed from the hot-phosphoric acid bath in the Footner's process, the plates, etc., dry rapidly and carry a protective dull grey phosphate film. The treated components can be transferred directly into the phosphating solution. The Footner's process cannot not be used in case of phosphoric acid pickling or zinc phosphating whereas it can be used only hydrochloric or sulphuric acid pickling.

10.2.2.4 Testing of de-rusting chemicals

To achieve best results it is necessary to test the de-rusting solution regularly and maintain it at the recommended strength. This involves a simple titration with a standard solution of caustic soda, using bromocresol green as indicator.

The de-rusting chemical progressively builds up iron as a result of the pickling reaction. The symptoms of excessive iron build up are an increase in processing time in spite of the bath strength being normal, or difficulty in rinsing the work properly or both. The point at which these symptoms may arise is not definite (being influenced by the strength of the bath) the amount of rust or scale present on the work, and the effectiveness of the rinse available. Usually from 7 to 10 percent iron may be tolerated, but above this the bath is spent, and should be discarded. The amount of iron present may be found by titrating 1 ml of the solution against N/10 potassium permanganate in the presence of sulphuric acid.

Percent iron (w/v) in the bath = $0.56 \times$ burette reading.

10.2.3 Electrochemical Treatment Method

It is possible to remove rust and scale by the following electrolytic methods:

- a) *Cathodic treatment in acid solution* — Removal of rust and scale may be accelerated as compared with ordinary pickling. Acid is economized and attack on the metal is reduced, but hydrogen embrittlement may be serious. The following composition is recommended:

i) Saturated citric acid solution	Approximately 20°C
ii) Sulphuric acid solution	(4 percent v/v)
Anode	Carbon or lead
Cathode	Test specimen
Cathode current density	20 A/dm ²
Inhibitor	A suitable organic inhibitor
Temperature	74°C
Duration	3 minutes
- b) *Cathodic treatment in alkaline solution* — Hydrogen embrittlement is less in this case than that of acid solution and the following solutions are used (current 1 A/dm²):
 - i) 8 to 10 percent sodium hydroxide, and
 - ii) 10 percent ammonium citrate.

- c) Anodic treatment may be carried out in either acid or alkaline solutions. Passivating conditions are established and oxygen (not hydrogen) is produced at the surface. Hydrogen embrittlement is usually avoided but there is a slight risk, especially with highly stressed parts, of hydrogen being formed in the acid process when the current is switched off and while the work is being removed from the bath. Anodic treatment produces smut on certain steels which should be removed by brushing and washing before phosphate treatment.
- d) *Examples:*
- An anodic process for parts entirely of steel based on sulphuric acid solution (sp gr 1.22) used at a temperature not exceeding 25°C in a lead tank with lead cathodes, with a high anodic current directly maintained on the steel surfaces (not less than 10.8 A/dm²).
 - An anodic alkaline process for parts entirely of steel based on a solution of caustic soda containing cyanide to facilitate removal of rust.

11 REMOVAL OF MISCELLANEOUS RESIDUES (HEAT-TREATMENT SALTS, FLUXES AND SWEAT RESIDUES)

11.1 Heat Treatment Salts

Heat treatment salts may contain the water-soluble cyanides, carbonates, chlorides or fluorides of sodium or potassium, with or without less readily soluble barium chloride. To avoid corrosion they shall be completely removed by washing in clean hot water. This may take from 5 minutes for articles of simple shape, up to 1 hour for those that have crevices or are coated with salts containing barium chloride. After removing the soluble salts in this way it is desirable to rinse the articles in a second tank of clean hot water to remove residues of contaminated wash water.

A help in removing salts of barium is to soak in hot water as above, transfer for a few minutes whilst hot to a cold solution containing 10 to 30 percent hydrochloric acid and an inhibitor swirl in cold running water and finally rinse in clean hot water. This acid treatment should be used if any cyanide remains on the articles.

11.2 Fluxes

Fluxes used in soldering, brazing or welding with the exception of certain materials (like rosin flux) require complete removal in order to avoid corrosion. This applies particularly to those containing hygroscopic chlorides or fluorides, or both. They may be removed by thorough washing the articles in water, for example, by immersing them in clean boiling water for 5 to 10 minutes, and if necessary, by dipping in dilute sulphuric acid additionally scrubbing with a bristle brush may be required to remove encrustations. The removal of flux residues

from article which have been brazed with a borate flux may be assisted by soaking in dilute sulphuric acid before thoroughly washing and scrubbing in water. Fluxes of an organic nature should be removed by appropriate degreasing methods. Where a chloride flux is compounded with a greasy medium, as with certain soldering fluxes, a degreasing treatment is required prior to water-washing. Particular attention should be paid to flux residues in crevices or seams.

11.3 Sweat Residues

Sweat residues left on surfaces may cause objectionable rusting or staining, particularly of highly finished bare steel. It is easy to avoid such contamination by using gloves, and, in particular, by not touching highly finished surfaces. If contamination has occurred, however, it should be removed without delay because the corrosion may proceed rapidly. These residues are not removed by petroleum or chlorinated solvents, but they are removed in aqueous processes such as alkaline cleaning, in addition, methanol to which 5 percent water has been added and certain oil-in-water emulsions have sweat removing properties. Special sweat-removing processes are not common in actual practice and for this reason are not detailed here.

SECTION 2 PHOSPHATING PROCESS

12 PHOSPHATE TREATMENT

12.1 The method of the phosphate treatment is to be to the satisfaction of the Inspecting Officer. The phosphate treatment shall be carried out strictly in accordance with the operating instructions issued by the proprietors of the process.

12.2 The bath concentration, temperature, and immersion time for parts shall be such that phosphate coating meets all requirements of specification.

12.3 The equipment shall be constructed of materials resistant to the action of the phosphating solutions and free from copper alloy fittings or brazing in contact with solution.

12.4 Phosphatized parts shall not be allowed to dry between the phosphatizing solution treatment and the subsequent cold running water rinse. Fog sprays or other means for prevention of drying shall be provided.

12.5 Control of the chemical content of the phosphate coating baths shall consist of determination of free acid, total acid, and ferrous iron. Analysis for free and total acid should be made just prior to the processing of each lot or every two hours, whichever is less frequent. The ferrous iron contents should be analysed just prior to the processing of each lot or weekly, whichever is less frequent.

12.6 Care should be taken at all stages to prevent contamination of the surfaces being treated by touching with bare hands, splashing with undesirable liquids or the condensation of moisture on components after drying, etc.

12.7 Ageing of Phosphating Solution

The freshly made up solution tends to produce coarse coating on the first few loads of work processed. Although this condition soon disappears and uniform coating is obtained, this may be avoided by ageing of the solution at the first stage which will later give consistent coating as and when the baths are used. The solution may be aged by placing a quantity of scrap iron or mild steel plate (roughly equivalent to one work load) in the bath for requisite duration and temperature prescribed by the process in use.

13 RINSING

13.1 After phosphating thorough rinsing with water is necessary in order to remove soluble salts which would otherwise tend to promote blistering under a paint film. Care should also be taken to ensure that the water supply itself is sufficiently free from harmful salts. Experience has shown that a water supply is potentially injurious if it exceeds anyone of the three following limits:

- 70 ppm total chlorides and sulphates (calculated as $\text{Cl}^- + \text{SO}_4^{2-}$),
- 200 ppm total alkalinity (calculated as CaCO_3), and
- Maximum of 225 ppm of (a) and (b) together.

13.1.1 Improved corrosion resistance and reduced tendency to blistering may be obtained by treating the final rinse with chromic acid or dichromate solution. After treatment by an unaccelerated process, the components shall be thoroughly rinsed in cold or hot water and then in hot dilute dichromate solution. In the case of an accelerated process, the components shall be rinsed thoroughly, first in running cold water and then in hot water at a minimum temperature of 75°C and shall be rinsed finally in hot dilute dichromate solution.

13.1.2 The chromate rinse shall consist of a solution

of chromic acid or alkali metal chromate or alkali metal dichromate, or a mixture containing approximately equal parts of chromic and phosphoric acids. The concentration of the solution shall be as given in Table 5 depending on the nature of the phosphating coating and of the sealing coat.

NOTE — Where a water rinse is used to avoid undue accumulation of the treatment chemicals in the rinsed water, the water shall be discarded when its acidity, expressed as ml of N 10 sodium hydroxide solution (4.0 g NaOH per litre) required to neutralize a 50 ml sample, exceeds 1.0 ml. The sample shall be taken after stirring the water and shall be cooled down before titration in which phenolphthalein shall be as indicator and the permanent pink colour shall be taken as the end point.

13.1.3 The grades of phosphoric and/or chromic acid used in the preparation of chromate rinse as also the quality of water shall be of such purity that the provision relating to freedom from corrosive residue shall be met.

13.1.4 In certain cases rinsing may be dispensed with, by agreement between the concerned parts, after unaccelerated phosphate treatment in the case of free-draining articles that contain neither pockets nor crevices and are to be sealed with an oil or non-drying coating, but blistering of paint due to local concentration of solution in seams and crevices may occur. Rinsing is generally applied, regardless of the type of phosphate process employed. For an accelerated process, where a water-soluble stain is to be applied a chromate rinse is unnecessary.

14 ADDITIONAL PRECAUTIONS TO BE TAKEN BEFORE OR AFTER PHOSPHATING

14.1 Treatment of Creviced Components

Components with fold, seams or crevices shall receive special attention to ensure the removal of oil or grease before phosphating. Care shall also be taken to ensure that the treatment chemicals are removed from folds, seams or crevices particularly when an accelerated phosphating process has been used.

Table 5 Concentrations of Chromate Solution

Sl No.	Nature of Phosphate Coatings and of Sealing Coat	Concentration in Terms of CrO_3^1 per 1 000 Litres	
		Min, kg (3)	Max, kg (4)
(1)	(2)		
i)	Phosphate coatings of all classes to be sealed with paint, varnish or lacquer	0.125	0.50
ii)	Zinc phosphate coatings to be sealed with oil or grease	0.125	2.50
iii)	Manganese and/or iron phosphate coatings to be sealed with oil or grease	0.125	5.00

¹The substitution of equal weight of phosphate acid for up to one half of the chromic acid is permissible.

14.2 Treatment of Composite Articles

In general, the advice of supplier of the phosphating material should always be sought regarding the treatment of composite articles because:

- a) contamination of the bath due to solution of non-ferrous metals may affect the phosphating reaction; and
- b) the phosphating solution may attack one or more of the component metals unduly, for example, through galvanic corrosion set up by contact of dissimilar metals.

14.2.1 Composite articles made up of ferrous and non-ferrous parts shall normally have their ferrous parts phosphated before assembly into the article. Exceptions are permissible in the case of composite articles contaminating zinc, provided that the phosphating zinc base solution does not cause an undesirable attack on the zinc surface.

The phosphating of composite articles containing copper-base material shall be limited to those cases where the copper-base material does not constitute more than 10 percent of the total surface, and where it is unlikely that the phosphating solution will penetrate the joints between the gel and the copper-base material. Tests shall be carried out to ensure that there is no undesirable attack on the copper-base material and that the phosphating solution does not become unduly contaminated with copper. In addition, checks shall be made that the presence of copper in the phosphate coating has no undesirable effect, such as interfering with the hardening of the paint, if paint, is to be applied. It is preferably to seal the copper components by stop off composition so that these surfaces do not come in contact with phosphating solution.

14.3 Stress Relieving Before Phosphating

14.3.1 General

The relieving of internal and deleterious surface stresses by a suitable heat treatment aids resistance to sustained load and fatigue failure. As a rule, internal stresses developed during quenching are relieved at temperatures of 400°C and above, and surface stresses developed during mechanical finishing operations can be reduced by heating at temperature of 200°C and above. The relief of beneficial surface compressive stresses introduced during production for example, by shot-peening or surface rolling should, however, be avoided. Stress relieving should be applied before cleaning, although parts may, if necessary, be degreased before heat treatment. The stress relieving treatment may be omitted for maraging steels which have been aged after final matching.

14.4 Severely Cold Worked Steel Parts

Severely cold worked steel parts may require stress relieving. As a guide, the parts should be maintained for 30 min at the highest temperature which can be applied without effecting their strengths.

14.5 Parts Made from Steels of Tensile Strength in the Range 1 000 N/mm² to 1 400 N/mm²

Parts made from steel of minimum specified tensile strength of not less than 1 000 N/mm² (or of equivalent surface hardness) nor more than 1 400 N/mm², which have been ground or subjected to heavy machining after final tempering should be stress relieved at a temperature between 130°C and 230°C (preferably near the higher end of this range) for not less than one hour, or to just below the tempering temperature for 5 to 30 min. Any stress relieving treatment should be applied after thorough degreasing and the subsequent complete removal of the degreasing medium before heating. Parts which have been treated in order to introduce beneficial compressive stresses into any parts of the surface, for example, by shot-peening or cold working, should not be heated above 130°C.

14.6 Parts Made from Steels of Tensile Strength Over 1 400 N/mm²

The standard stress relieving consists of heating the parts to the highest temperature possible within the limit imposed by the tempering or ageing temperature or by any susceptibility of the steel to temper brittleness, and in any case not less than 200°C. The period at temperature should be not less than either 18 h at 200°C, 6 h at 250°C, 2 h at 300°C, or 1 h at 400°C or above, followed by cooling in still air. Case hardened parts which are not be phosphated on the hardened may, however, be stress relieved at only 300°C for 6 h, as a higher temperature may reduce the hardness of the case to an unacceptable degree.

14.7 Spring or other components subject to flexing made from steels with a specified minimum tensile strength of 100 kgf/mm² shall not be processed in compression or tension, that is, while the component is Hunder applied stress. Unless otherwise agreed, the phosphating process used in such cases shall be of the accelerated (but copper free) type and the pointage of the bath shall not exceed 30.

14.7.1 In the case of springs or where soldered joints are employed it may be necessary to keep the heat treatment below 130°C; otherwise the treatment given below may prove suitable:

Specified Range of Tensile Strength kgf/mm ²	Conditions of Temperature		Heat Treatment Duration of Heating, h, Min
	Min °C	Max °C	
100 to 140	130	200	1
Over 140	150	200	4

14.8 The above standard stress relieving procedure should not applied to all parts which have not been given a mechanical treatment to introduce beneficial compressive stresses into the surface, for example, by shot-peening or surface rolling. Standard stress relieving prior to treatments given to introduce

beneficial compressive stresses into the surface is recommended and, if applied, should not be repeated after the mechanical treatment. If no prior stress relieving is given, then parts given a mechanical treatment all over should not be stress relieved after treatment and parts given a mechanical treatment over only part of the surface should be subsequently stress relieved by the standard procedure but at a temperature within the range 200 to 230°C.

15 STAINING

Where necessary and appropriate, the phosphated components shall be stained by water stain. Water staining consists of treatment in a solution of water soluble dye. Water stain shall be applied after rinsing. This only improves the appearance and has no corrosion resistance property.

16 DRYING

16.1 After the final rinsing, the components, shall be dried thoroughly (preferably force-dried), special attention being paid to the parts that contain pockets or crevices. Precautions shall also be taken to avoid local accumulation of solid residues such as may be formed on the surface when hard water is used for rinsing.

16.1.1 Compressed Air for Drying Purposes

The compressed air should be dry and free from oil

and dirt. The presence of moisture in compressed air may be readily detected by permitting the air to blow on a polished metal part at room temperature, and observing any condensation. The pressure of air should not be above 6.3 kgf/cm².

Moisture traps should be placed at the lowest point in the air delivery pipe and as close as practicable to the jet. Air filters combined with (or separate from) the moisture traps should be fitted. Traps should be drained frequently during operation and filters cleaned regularly. Air receiver tanks usually have a relief valve or blow-off cock at the bottom; it should be opened when the receiver is not in use and left open until the compressor is used again. On re-using the compressor, air should be allowed to blow through the relief valve for a minute to blow out any condensed moisture in the receiver, the valve closed and air jet tested at the delivery end to detect any moisture present. This is necessary because moisture may be present within the air line when the plant is idle and as a result of the process of compression.

16.1.2 Use of Water-Displacing Fluids

These materials are volatile solvent which by the addition of special substances are unable to displace water from metal surfaces. The articles to be dried are immersed in the liquid without agitation and the water displaced falls to the bottom of the tank and may be drained away, the design of a dip-tank for automatic drainage of the water is shown in Fig. 3.

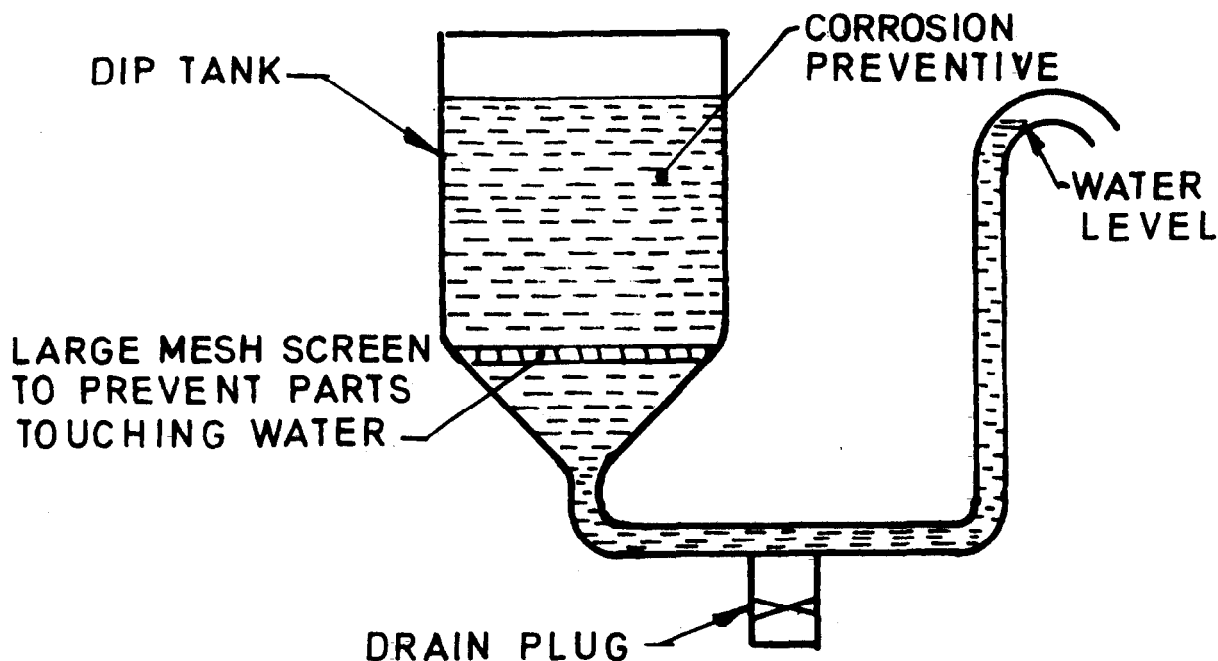


FIG. 3 DIP TANK FOR WATER DISPLACING FLUID

16.1.2.1 A thin protective film of organic material is left as residue after the above treatment. If this residue is not compatible with the final finishing treatment of oils, paints, grease, etc, the residue should be removed by petroleum solvents.

16.1.3 *Drying by Wiping*

This method of drying should be used only if no other method is available. Cloth or waste used for wiping should be clean and dry, and should not leave lint on dried surfaces. The process should be carried out in two steps, the first cloth should leave an apparently dry surface, and this should then be wiped with a second, clean, dry cloth to ensure a thoroughly dried surface.

17 REPAIR OF DAMAGED PHOSPHATE COATING

In some cases, for example, in coach-building practice, the steel sheets or other parts after hot-dip phosphating may have to be welded and in the process, the phosphate coating gets damaged. Cold phosphating normally used to repair the damage after cleaning the welded areas with wire brush.

18 TESTING AND MAINTENANCE OF PHOSPHATING SOLUTION

18.0 The phosphating solution shall be regularly tested once a day or in high volume production lines once a shift, to ensure that consistent coatings are deposited. Failure to do this results in patchy or powdery coatings of low coating weight, with a corresponding reduction in corrosion resistance.

18.1 Phosphating solutions are either operated with the deliberate addition of oxidizing agents referred to in the trade as 'toner' or 'compensating chemical' or are allowed to accumulate soluble iron salts as a result of the dissolution of metal in the initial stages of the phosphating process. The former type of solutions operate 'iron free' with a positive content of the oxidizing agent.

Phosphating solutions are generally maintained in the industry by a pointage titration, and a toner or iron titration. Details of carrying toner test are given in Annex A.

18.1.1 *Determination of Iron Content*

This method is used to determine the iron content of those pre-treatment baths which are operated with certain limits of ferrous iron to get optimum results. The test method consists in titrating 10 ml of bath solution against N/10 potassium permanganate in the presence of sulphuric acid to a pink end point.

Iron, percent (w/v) = $0.056 \times \text{burette reading}$

19 FINISHING

19.1 The various types of supplementary finishes,

quality of materials to be used, methods of application, etc, are given below:

- a) *Paint* — The component shall be painted with one of the paint (given in IS 2074 or any other suitable paint. Two coats of paint should be applied, preferably by spraying, the second not being applied until the first has thoroughly set.
- b) *Stoving Paint* — In the case of stoving paint given in IS 2075 the first coat may be 'flash-dried' and the second shall be stoved at the temperature and for the period appropriate to stove and to the paint, to ensure that the paint is fully stoved.
- c) *Stoving Lacquer* — The component shall be coated with clear stoving lacquer of the type most appropriate to the store concerned and to the method to be used for applying and stoving the lacquer. When coated, the component shall be stoved at the temperature and for the period appropriate to the store and to the lacquer to ensure that the lacquer is fully stoved.
- d) *Grease* — The component shall be treated with grease, mineral jelly or preservative mineral jelly-beeswax (9:1) for soft thick film.
- e) *Lanoline*
 - i) The component shall be dipped in a solution of lanoline in white spirit maintained in the proportion of 1:9. After dipping, the component should be allowed to drain in a current of warm, dry air.
 - ii) The component shall be coated by dipping in a homogeneous solution of 35 percent lanoline and 65 percent white spirit for soft thin film. The component shall be allowed to stand until the coating has set. Setting of the lanoline solution is aided by directing a current of warm, dry air over the component. The solution shall be maintained in the correct proportion by adding white spirit as necessary to replace that lost by evaporation.
 - iii) *Stain* — The component shall be immersed in the stipulated water-stain solution, or in stain, varnish and black (spirit stain) for the period recommended for that particular process. Spirit stain consists of shellac dissolved in methylated spirit and spirit soluble dye; methylated spirit free stain may also be used. This shall be applied after drying. It gives additional corrosion resistance due to the film of shellac deposited on the phosphated surface. The component shall be dried thoroughly by stoving or force drying as appropriate, any loose powder or deposit removed.

NOTE — Water stain may be heated, if necessary. Spirit stain should not be heated.

- f) *Oil* — The component shall be dipped or sprayed with suitable oil or lubricating oil (internal-combustion-engine preservative). After dipping all surplus oil shall be allowed to drain off and the oil film allowed to dry in warm dry atmosphere.
- h) *Linseed Oil* — The component shall be wiped or brushed lightly with a solution of linseed oil, refined, in white spirit or turpentine in the proportion 1:9. The component shall be allowed to dry, after draining if dipped, in warm air or shall be oven dried.

NOTE — Wiping for removal of surplus oil is not allowed.

- g) *Lacquer, Clear* — The clear lacquer shall be applied by spraying or dipping when the component is sufficiently hot.

20 INSPECTION AND TESTING

The procedure for testing and inspection of phosphate coating shall be as given in IS 3618.

ANNEX A

(Clause 18.1)

TESTING OF TONER CONTENT OF PHOSPHATING SOLUTIONS

A-1 This test is meant for only those baths that are operated with the positive presence of toner crystals.

A-1.1 The toner content of the bath is determined by titrating (*see A-1.1.2*). However, before this titration test is carried out, it is essential, to check the positive presence of toner in the bath. This is done by the starch-iodide-paper test (*see A-1.1.1*). If the starch-iodide paper remains white indicating that there is no toner in the bath, then toner has to be added as a 25 percent solution in small increments till the correct quantity of toner is present. When this condition is reached, the starch-iodide paper turns blue. The toner content of the bath may now be determined by titration against N/10 potassium permanganate.

A-1.1.1 Test with Starch-Iodide Paper

The following changes in the starch-iodide paper takes place when it is dipped in the Granodine bath:

<i>Toner in the Bath</i>	<i>Colour of Sodium Iodide Paper</i>
Insufficient or none	Remains white
Correct	Pale mauve to blue
Excessive	Blue-black to black

A-1.1.2 Test by Titration

Titrate 50 ml of bath solution against N/10 potassium permanganate in the presence of sulphuric acid to a pink end point.

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